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DEPARTMENT OF COMMERCE

TECHNOLOGIC PAPERS OF THE BUREAU OF STANDARDS

S. W. STRATTON, DIRECTOR

No. 176

SLUSHING OILS

BY

PERCY H. WALKER, Chemist

LAWRENCE L. STEELE, Associate Chemist

Bureau of Standards

OCTOBER 14, 1920



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I. INTRODUCTION

Recently the Bureau of Standards has had numerous inquiries regarding information as to methods of protecting from corrosion metal in storage for rather long periods. These requests generally refer to the protection of metal articles which are of such a nature as not to allow the application of metallic coatings, such as galvanizing, or of vitreous enamels, paints, or varnishes. It is obvious that with bearing parts of machinery, coatings such as the above, which form dry and hard films and can not be easily removed, are not suitable. Our attention is therefore confined to a consideration of protective coatings which remain in a soft condition so that they can be easily removed at any time.

Regarding the length of time these coatings will afford adequate protection, there is little information available. It is well known that good oil paint will protect steel for several years when exposed to the weather, and that a good varnish will protect steel for several months. The protection afforded by paint and varnish to articles when kept indoors is very much greater than when exposed to the rays of the sun. While no data based on experiments are available, it is safe to assume that coating with a good grade of varnish and storing protected from the weather may be expected to preserve metal for several years. Paint under similar conditions may be expected to preserve metal for a considerable number of years. As mentioned above, however, these two types

of materials are not applicable for use on the bearing parts of machinery, or on many metallic articles which must be protected from corrosion. Such bright metal is protected by coating it with a layer of nondrying grease, usually known as slushing oil or slushing compound.

II. NATURE OF SLUSHING OILS

Slushing oils or slushing compounds are of a varied nature, sometimes being straight mineral greases (petroleum residuum, such as petrolatum), sometimes mixtures of mineral and animal greases with or without the addition of soap (such as lime rosin or lime fatty acid soaps), finely divided mineral matter, and volatile solvents. Sometimes the petroleum base is mixed with rosin or rosin oils. For example, certain branches of the Army used for a number of years on rifles and guns a compound composed of petrolatum mixed with not over 20 per cent rosin. Mineral oils as fluid as light spindle oils have been used for this purpose, but from the discussion which will follow it will be seen that such thin products are of very doubtful value except for very limited and particular purposes.

From one of the primary requirements of such materials, that they should be easily removed when desired, it follows that it is impossible to get a material of this nature which can not be rubbed off, although there is a very marked difference in ease of removal by abrasion.

III. DESIRED PROPERTIES OF SLUSHING OILS

An ideal slushing compound is one which can be easily applied to all kinds of metal surfaces by a variety of methods. It should coat the surface with a sufficiently thick and impervious film to exclude moisture and air (to prevent rusting), should remain in position for an indefinite length of time and yet be completely removable from the surface without undue labor. The material should itself have no corrosive action on any kind of metal. It is believed that the above statements give the essential requirements of a slushing compound. In addition, however, it is very desirable that the coating formed should be sufficiently transparent so that inspection to detect beginning of corrosion can be made without cleaning the article.

Metal articles which are absolutely protected from moisture, air, and other gases will never corrode. It therefore follows that any coating which will permanently exclude moisture, air, and

any other gases commonly met with in the atmosphere, and which itself has no corrosive action must of necessity protect the metal from rust. There is, of course, no material which will to the fullest extent meet all of the above requirements, but for practical purposes it is believed that a number of materials of the varied composition indicated above answer very well for this purpose. No such materials in very thin films are absolutely impervious to air and moisture, hence very fluid compounds that leave only very thin films on the metal will not give as good protection as the same character of compound of a thicker consistency.

IV. OBJECTION TO HARD AND FAST REQUIREMENTS AS TO COMPOSITION OF SLUSHING COMPOUNDS

It follows from what has been previously said regarding variation in the composition of slushing compounds that any definite limitation on composition of the material desired may exclude from consideration perfectly satisfactory materials which do not meet the particular requirements as to composition and will thus tend to increase price.

In some cases we have noticed composition requirements which not only limit in an unjustifiable manner the choice of material for making a satisfactory article, but in some cases are so worded that it is practically impossible to secure any material which will meet the requirements. For example, we have seen specifications which called for practically pure petroleum products free from even a minute trace of acid. This clause was probably introduced because the acids most commonly thought of, mineral acids and the strong organic acids, are corrosive to metal, and such substances should not be present in any material intended for protecting metal from corrosion. On the other hand, some resin acids have practically no effect on the common metals, and resins containing these acids are frequently very valuable additions to slushing oils, producing products which have superior physical properties to the original petroleum base. Even without the addition of such resins, the petroleum base will never be free from small quantities of acid. It is well known that all petroleum products when exposed to light and air develop more than traces of acid (Waters, B. S. Scientific Papers, No. 153, and Technologic Paper No. 73).

Another requirement which has appeared in certain specifications and which appears to have originated in erroneous application of certain well-known facts is that the material should con-

tain chemicals that are known to be rust inhibitors. It would, undoubtedly, be a good thing to secure a slushing oil to which actual rust-inhibiting properties had been given. Unfortunately, there is no known method of so altering these materials as to render them anything but at best inert in themselves. Regarding the inhibition of rust, it is of course well known that certain chemicals under particular conditions produce a so-called passive condition of iron, leaving the surface in a condition which resists rust for limited periods of time or resists rust under particular conditions of exposure. The well-known example of treating iron with very concentrated nitric acid might be mentioned, and of course it is ridiculous to consider that nitric acid, because under certain conditions it renders iron passive, would be a good material to add to a material for protecting iron from rust.

Iron and steel immersed in water solutions of strong alkalis will not rust as rapidly as when immersed in pure water. The water solutions of soluble chromates are among the best known rust-inhibiting substances, and if iron or steel is kept immersed in an aqueous solution of soluble chromate, it will resist rust for a much longer time than the iron immersed in water. This property of soluble chromates appears to have been the basis of the idea that rust-resisting chemicals could be satisfactorily added to slushing oils. One can not predict the action of chemicals when incorporated in any way with an oily medium from the action of these same chemicals when in water solution, as is shown very clearly by some very interesting experiments which were conducted on paints.

It had been found that some paint pigments, when suspended in water, had a marked influence on the speed with which iron and steel immersed in the water rusted. A large number of paint pigments were tested in this manner, and it was found that they could be divided into three classes—those in which the iron or steel rusted very much slower than in pure water, which for sake of convenience were called water inhibitors; those in which the iron or steel immersed in water containing pigment rusted in about the same degree as in pure water, which were called indeterminates; and those in which the iron or steel immersed in water containing the pigment rusted distinctly more rapidly than when in pure water, which were called water stimulators. This fact attracted so much attention that very extensive tests were made to ascertain whether these same pigments in an oily vehicle would behave in the same manner as they did in suspension in water. These

tests were carried out under the direction of two committees of the American Society for Testing Materials.¹ The committees of this society carefully inspected these tests, which extended over a period of more than five years, and in rendering their final report made the following statement:

The summarized results indicate that the water test as at present developed can not be depended upon to give accurate information as to the protection which pigments will or will not afford metal structures when such pigments are applied in oil media. The summarized results also indicate that the arbitrary designations, inhibitive, indeterminate, and stimulative, as referring to the results of the water test, are not always applicable to the same pigments when used with linseed oil for protective coatings.

A single illustration taken from tests on numerous pigments by the above-mentioned committee will convince one that this conclusion was entirely justified as respects paints. Among the pigments selected were two samples of Prussian blue, Nos. 44 and 45. No. 44 Prussian blue was intentionally selected as one which in the preliminary water test acted as a stimulator of corrosion. No. 45 was one which under the water test acted as an inhibitor of corrosion. This Prussian blue No. 45 contained some chromate. In June, 1913, before the conclusion of this test, it was necessary to remove some of the panels, and the committee selected for discontinuing a number of panels which at that time were considered as having failed; among them was the inhibitive Prussian blue No. 45, whereas the stimulative Prussian blue No. 44 was not included in this list. It is probable that these observations were never carefully considered by the people who have advocated the use of so-called rust-inhibiting chemicals in slushing oils. So far as the writers know, all manufacturers who have attempted to produce slushing oils containing rust-inhibitive chemicals have prepared a product which was essentially an emulsion of a water solution of sodium or potassium chromate in a straight petroleum residue base. A careful consideration of the nature of this mixture would lead one to the conclusion that the water solution of the chromate could not possibly have any beneficial effect. Such a material when completed would consist of a large quantity of heavy petroleum product with minute globules of water solution of chromate distributed through it. These globules do not come in contact with the metal on which the slushing compound is coated and hence can have no effect as rust inhibitors. Soluble chromates

¹ A. S. T. M. Proceedings, IX, p. 106, 1909; X, p. 75, 1910; XI, p. 192, 1911; XIII, p. 369, 1913; XIV, Part 1, p. 259, 1914; XV, Part 1, p. 222, 1915.

frequently cause serious injury to the skin, and some users of slushing oils containing chromates have been obliged to abandon them on this account.

V. LABORATORY TESTS ON SLUSHING COMPOUNDS

A few months after the United States entered the Great War, the Bureau of Standards started to receive samples of rust preventives from branches of the War Department. This material was intended for use on guns, rifles, and small arms and was purchased on a somewhat indefinite specification. At the request of the War Department the following tests were made on these samples: (1) Test for mineral acid; (2) test for rosin; and (3) an adhesion test.

A test was made for mineral acid by heating some of the sample with distilled water and qualitatively testing the water portion for acid in the usual way. Rosin was detected by the Liebermann-Storch reaction. The adhesion test was performed as follows: The sample was smeared on both sides of a tared copper plate 2 inches square and 0.0063 inch in thickness. This plate was then suspended by one corner in an oven and maintained at a temperature of 65° C (149° F) for 30 minutes. After cooling, the plate was weighed. An increase in weight of the plate of not less than 65 and not more than 85 mg was required.

The above tests probably gave very little information as to the merits of a given slushing oil. From results of work done later, it is believed that no adhesion test, where the film of oil remaining adherent to a test plate under set conditions is weighed, can be relied upon to differentiate between good and bad rust preventives.

Practically all the samples of slushing oil examined for the War Department by the methods outlined above were straight mineral oils of the consistency of petrolatum and did not contain thinners or mineral matter.

During the summer of 1918 the Bureau of Standards received at different times various samples of rust preventives from the War Department with a request in each case that certain extravagant rust-inhibiting qualities claimed by the manufacturer be investigated.

These compounds were analyzed in so far as it was possible and in addition it was thought best that some comparative test be made, using one of the regular samples of rust preventive in use by the War Department as a standard of comparison. To

this end exposure tests on bright steel plates were made. These exposure tests have proved to be of very great value in the evaluation of rust preventives, and the details of the method will be explained at this point: Bright cold-rolled steel of No. 26 gage was cut into pieces measuring 4 by 6 inches and carefully cleaned by the use of cotton waste and benzene. The sample of rust preventive was then applied to these plates either by dipping, brushing, or smearing on with a spatula, according to the nature of the material. It was found convenient to tack the plate to a board before applying the slushing oil, so that the coated plate could be carried without danger of disturbing the film of rust preventive. The coated plates were allowed to stand for one day in the laboratory in an upright position and were then exposed outdoors at an angle of 45° , facing south. They were inspected at intervals of every few days and a record kept of the condition of the various plates.

These exposure tests proved to be of great value in the comparison of a number of samples. The length of time before rust appeared on the plate was influenced very largely by the kind of weather and the season of the year, so that the tests were only comparative. These first exposure tests indicated one fact which has been confirmed in nearly all cases by later series of tests, namely, oils of thin consistency give very poor protection to steel exposed to weather as compared with oils of greater body and hence greater adhesiveness. This is a conclusion which seems most logical.

At the time when these first exposure tests were made, an interesting phenomenon was noted. A compound was being examined, the manufacturers of which claimed it possessed rust-inhibiting qualities. This material was found to be essentially an emulsion of a small amount of a water solution of a chromate in heavy mineral oil. The rust-inhibiting properties of chromates in slushing oils have already been discussed on page 6. When this sample was applied in the usual way to bright steel plates, either by smearing on the compound from the original container or else by applying the melted material, and the plate allowed to stand in an upright position in the laboratory, it was noted that the film of rust preventive slipped down the surface of the metal. The material did not melt on the panel and run off, but the whole film moved down the plate like a glacier and left the surface of the metal bare. At first it was believed that the slipping of this

particular rust preventive was due to the small amount of water present in the oil chromate solution emulsion. However, other slushing oils were found later which were free from water and which showed the same phenomenon of slipping from bright metal surfaces.

The cause for this slipping observed in cases of certain rust preventives has not been determined. It is readily seen that a property of slipping from bright metal is a very serious defect in a slushing oil. At the suggestion of the Bureau of Standards, certain Government specifications have included the following clause or a similar requirement:

When a highly polished steel plate 3 by 5 inches, coated with rust preventive, is allowed to stand on the shortest side, with the longest side vertical, for one week, there shall be no slipping of the rust preventive and no portion of the steel surface shall be exposed through movement of the protective coating.

The above clause, it was believed, would eliminate those rust preventives which possessed the serious defect of slipping.

After the signing of the armistice different branches of the War Department became concerned in the problem of storage of guns, airplane engines, machines, etc. The Bureau of Standards received numerous requests for information as to the best methods for the protection of metal from corrosion during storage for long periods of time. Mimeographed sheets entitled "Notes on Protection of Metal in Storage" were prepared and issued on January 4, 1919. In these notes tentative specifications were outlined for three different types of rust preventive: Brushing consistency, dipping consistency, and an opaque compound for special uses.

These specifications did not dwell upon composition of material, but an attempt was made to include certain laboratory tests which, in conjunction with the comparative exposure tests, should give a basis for the evaluation of different samples of slushing oils.

A new adhesion test was prescribed which differed considerably from the War Department method previously described on page 8. This modified adhesion test was as follows:

(a) Two polished copper plates 5 by 5 cm and not more than 1.0 mm thick, will be weighed and then coated on both sides with the slushing oil. One of these plates will be suspended by one corner for 30 minutes, in an oven at 75° C, the other in an oven at 65° C. The two plates will then be weighed. The oil remaining on the plate heated at 65° C shall weigh not less than 60 mg and the oil remaining on the plate heated to 75° C shall not be less than three-fourths as much as that remaining on the plate heated to 65° C.

(b) When applied to a polished metal plate 10 by 15 cm and the plate then held in a vertical position at any temperature between 15° and 35° C for 5 days, the coat of slushing oil shall remain adherent to the metal, shall not slide appreciably from the top edge of the plate nor leave any portion of the surface.

The clause which required that 75 per cent as much material remain on the plate at 75° C as remained at 65° C was designed to eliminate those rust preventives which rapidly lose their adhesive qualities with a slight increase in temperature. The weight of material required to remain on the plate after 30 minutes at 65° C was arbitrarily taken at 60 mg or more in the case of slushing oils of brushing consistency and 30 mg or more in the case of slushing oils of dipping consistency.

Experience with these adhesion tests has shown them to be of doubtful value in the examination of slushing oils. This point will be discussed in full further on.

In the question of acid content of rust preventives a very radical departure was made. Most slushing oil specifications required that "no acid be present." As already pointed out on page 5, pure mineral oils are likely to develop traces of acid on exposure to light and air. Furthermore, the detection of traces of acid in a petroleum is not certain by present methods. For these reasons it was not specified that no acid be present, but a requirement was added in the specification that "the rust preventive in question should not corrode bright copper, brass, nickel, or iron after five days." It is firmly believed that this requirement in regard to corrosion effectively excludes any slushing oils which contain harmful acid in quantity sufficient to cause trouble in actual use. Harmful acids which may be present in rust preventives cause stains to appear upon bright copper and brass in a comparatively short time, and therefore these metals act as sensitive indicators of the presence of corrosive substances in the slushing oil.

The exposure tests made on bright steel and already described in some detail are believed to be the most important of all the tests proposed in the new tentative specifications for slushing oil.

Since almost any rust preventive in use may be subject to exposure to the weather, the most vital property of a good slushing oil should be that it give an impervious and lasting film on metal, even when subjected to the action of rain, snow, and rapidly changing temperature. The great objection to exposure tests is the length of time that is needed to make them properly. Plates must be exposed for at least 30 days, and better for a space

of two months, in order to draw any valuable conclusions. The time of the year must also be taken into account, because hot summer weather with rain is much more severe on rust preventives than cold winter weather with snow and ice.

A few months after the circular "Notes on Protection of Metal in Storage" had been issued, one of the purchasing bureaus of the War Department sent in a large number of samples of slushing oil with a request that an examination of these materials be made to determine if possible which brands were best suited for rust prevention. There were 44 samples in all, and tests were begun at once, using the methods of test outlined in the specifications of the circular of January, 1919.

The 44 samples were first grouped into three classes: Class 1, the transparent materials of brushing consistency at temperatures above 10°C (50°F); class 2, the opaque materials of brushing consistency at temperatures above 10°C (50°F); class 3, the materials of dipping consistency at temperatures above 10°C (50°F).

Each one of the 44 samples was examined under all the laboratory tests given in the circular, and in addition each material was ashed and the percentage and nature of the ash determined, the acid number was determined, and a qualitative test for rosin was made in each case.

The results of the laboratory tests will be found in Table 1.

Exposure tests lasting for a period of 60 days were made on all the samples. The materials which flowed readily at room temperature were applied by flowing an excess of oil over the plate and allowing the surplus to run off. The materials which were semisolid at room temperature were applied in two ways: (1) One set of plates was coated by smearing on an excess of material as it came from the container; (2) another set of plates was coated with melted material. The plates were coated on July 18, 1919, and exposed to the weather on July 19, 1919. The weather conditions during the following 60 days were unusually severe. There were many hard rains with intervals of hot sunny days. Some of the materials which did not stand up well in the exposure tests might give good protection under less severe weather conditions, but the tests were intended to be severe. It is believed that a satisfactory rust preventive should stand up well on metal for at

least 60 days when exposed to the most severe weather conditions which may prevail at points where the material is to be put in service.

The results of the exposure tests upon the 44 samples after 60 days on the roof are given in Table 2.

In order to compare the results obtained in the exposure tests with the results in the laboratory tests, Table 3 was prepared. It must be kept in mind that several of the rust preventives of semisolid consistency were applied to the steel plates in the exposure test both as a paste and in a melted condition. Where different protection was given, depending upon the mode of application, that sample of slushing oil has been counted twice and the same compound may appear, for example, listed as both "fair" and "failure."

It is a fact worthy of note that nearly all the compounds of semisolid consistency gave the best protection in the exposure test when they were applied to the steel plate in a melted condition.

An examination of Table 3 shows that a strict interpretation of the laboratory tests given in the tentative specification of January, 1919, would have excluded many compounds which showed up well on exposure and would have passed at least three compounds which failed in the exposure test. For example, if the five compounds excluded in the laboratory tests because the resulting film was not removable with kerosene are not considered, we find that a strict interpretation of the adhesion and slipping tests alone would have excluded one compound rated as perfect in the exposure test, one compound rated as good, and eight compounds rated as fair, while two samples rated as complete failure and one sample rated as partial failure would have been accepted from the results of the laboratory tests alone.

The above comparison shows clearly that it was unwise to depend entirely upon laboratory tests outlined in the tentative specifications for decision in regard to acceptance or rejection of rust preventives.

TABLE 1.—Laboratory Tests on 44 Samples of Slushing Oils

B. S. No.	Slipping	Corrosion			Adhesion			Ash		Acid No.	Rosin	Film
		Steel	Copper	Brass	At 65°C	At 75°C	Per cent	Per cent	Contains			
55784	O. K.	O. K.	O. K.	O. K.	Mg	Mg	59.5	0.07	Fe.	27.7	Not detected.	Black opaque.
55785	Slipped slightly on steel; did not leave surface.	do.	do.	do.	57.6	34.2	78.4	.07	do.	.12	do.	
55786	O. K.	do.	Corroded.	Corroded.	50.1	46.3	92.4	.06	do.	41.9	Present.	Black opaque.
55787	Slipped badly	do.	O. K.	O. K.	83.3	64.3	77.2	.54	Na, Cr.	.19	Not detected.	
55788	do.	do.	do.	do.	60.1	59.7	99.4	.34	do.	.19	do.	Black opaque.
55789	do.	do.	do.	do.	83.6	45.4	54.2	.70	do.	.12	do.	
55790	O. K.	do.	do.	do.	50.3	31.1	61.8	.00	do.	(a)	do.	Black opaque.
55791	Slipped badly	do.	do.	do.	71.8	52.4	73.0	.01	do.	.06	do.	
55792	do.	do.	do.	do.	105.3	64.3	61.0	.67	Ca.	.12	do.	Black opaque.
55793	Slipped.	do.	do.	do.	76.2	39.6	52.0	.04	do.	.25	do.	
55794	O. K.	do.	do.	do.	268.0	264.4	98.6	.22	do.	.37	do.	Black opaque.
55795	do.	do.	do.	do.	28.0	27.6	98.5	.00	do.	.12	do.	
55796	do.	do.	Slightly corroded.	Slightly corroded.	134.4	97.0	72.0	.20	do.	.19	do.	Black opaque.
55797	do.	Rusted under film	O. K.	O. K.	32.2	27.2	84.5	.00	do.	.03	do.	
55798	do.	O. K.	do.	do.	678.6	668.6	98.5	.50	do.	1.99	do.	Black opaque.
55799	do.	do.	do.	Corroded.	30.4	30.0	98.7	.50	Mn, Ca.	7.2	Present.	
55800	do.	do.	do.	O. K.	146.2	110.8	75.6	.60	do.	5.4	do.	Do.
55801	do.	do.	do.	do.	2,025.0	1,979.0	97.6	2.85	Fe, Ca.	.31	Not detected.	
55802	do.	do.	do.	do.	106.2	85.2	80.4	24.0	Ba, Zn, S, SO ₄ .	.19	do.	Do.
55803	do.	do.	do.	do.	101.0	83.2	82.3	7.00	Pb.	.25	do.	
55804	Slipped.	do.	do.	do.	46.8	46.0	98.3	.03	Pb, Fe.	.12	do.	Dark brown opaque.
55805	O. K.	do.	do.	do.	348.2	294.2	84.5	18.00	do.	.68	do.	
55806	do.	do.	do.	do.	38.0	37.8	99.5	.24	Mn, Pb, Ca	13.6	Present.	Dark brown opaque.
55807	Slipped badly	do.	do.	do.	84.0	65.8	78.3	.00	do.	.12	Not detected.	
55808	O. K.	do.	do.	do.	89.4	71.4	79.8	.00	do.	.19	do.	Dark brown opaque.
55809	do.	do.	do.	do.	67.0	48.0	71.6	.00	do.	.03	do.	
55810	Slipped badly	do.	do.	do.	44.4	43.4	97.7	.01	do.	.03	do.	Dark brown opaque.
55811	O. K.	do.	do.	do.	47.2	47.0	99.5	.00	do.	.03	do.	
55812	do.	do.	do.	do.	52.8	51.8	98.2	.00	do.	.37	do.	Dark brown opaque.
55813	do.	do.	Corroded badly	do.	45.4	45.2	99.5	.00	do.	22.60	do.	

55814	do.	do.	O. K.	do.	50.8	49.2	96.8	.06	3.50	Present
55815	do.	do.	do.	do.	56.2	37.4	66.5	Not detected
55816	Slipped badly	do.	do.	do.	73.8	57.8	73.4	.00	.03	do.
55817	Slipped slightly	do.	do.	do.	70.4	70.4	100.0	.00	.00	do.
55818	Slipped badly	do.	do.	do.	75.5	57.0	75.5	.00	(a)	do.
55819	O. K.	do.	do.	do.	47.6	48.2	101.3	1.30	9.27	Set to touch.
55820	do.	do.	do.	do.	138.2	135.2	97.8	.00	16.2	Do.
55821	Rusted under film	do.	do.	do.	36.6	35.4	96.8	.00	.03	Not detected
55822	Rusted slightly under film.	do.	Slightly corroded.	do.	40.0	38.2	95.5	.00	.03	do.
55823	do.	do.	do.	Corroded	121.0	85.5	70.6	.00	12.6	do.
55824	do.	O. K.	Corroded badly	Corroded badly	181.2	144.4	79.7	.00	18.0	do.
55825	do.	do.	do.	do.	126.8	87.8	69.4	.00	16.4	do.
55826	do.	do.	do.	do.	125.0	104.8	83.8	.01	22.1	do.
55827	do.	do.	O. K.	O. K.	118.2	81.6	69.0	.06	.50	Opaque.

^a Slightly alkaline.

TABLE 2.—Results of Exposure Tests for 60 Days upon 44 Samples of Rust Preventive Received from the War Department on July 11, 1919

B. S. laboratory No.	Materials ^a	Appearance of first rust	Condition of plate after 60 days ^b
55784.....	L	After 2 days.....	Complete failure.
55785.....	L	After 35 days.....	Partial failure.
55786.....	L	After 2 days.....	Complete failure.
55787.....	S	do.....	Fair.
55787.....	M	After 22 days.....	Do.
55788.....	L	After 4 days.....	Complete failure.
55789.....	S	After 2 days.....	Partial failure.
55789.....	M	After 4 days.....	Fair.
55790.....	L	After 2 days.....	Complete failure.
55791.....	S	After 3 days.....	Partial failure.
55791.....	M	After 60 days.....	Good.
55792.....	S	After 46 days.....	Fair.
55792.....	M	After 27 days.....	Partial failure.
55793.....	S	After 46 days.....	Fair.
55793.....	M	After 26 days.....	Do.
55794.....	L	After 60 days.....	Good. ^c
55795.....	L	After 2 days.....	Complete failure.
55796.....	L	do.....	Do.
55797.....	L	do.....	Do.
55798.....	L	None.....	Perfect. ^c
55799.....	L	After 4 days.....	Complete failure.
55800.....	L	After 60 days.....	Good.
55801.....	S	None.....	Perfect.
55801.....	M	After 32 days.....	Fair.
55802.....	S	None.....	Perfect.
55802.....	M	do.....	Do.
55803.....	S	After 2 days.....	Partial failure.
55803.....	M	do.....	Fair.
55804.....	S	do.....	Partial failure.
55804.....	M	do.....	Complete failure.
55805.....	S	None.....	Perfect.
55805.....	M	do.....	Do.
55806.....	L	After 18 days.....	Complete failure.
55807.....	S	After 2 days.....	Partial failure.
55807.....	M	After 46 days.....	Fair.
55808.....	L	After 10 days.....	Complete failure.
55809.....	S	After 14 days.....	Partial failure.
55809.....	M	After 2 days.....	Do.
55810.....	S	do.....	Do.
55810.....	M	do.....	Do.
55811.....	S	do.....	Fair.
55811.....	M	After 18 days.....	Do.
55812.....	L	After 2 days.....	Complete failure.
55813.....	L	do.....	Do.
55814.....	L	None.....	Perfect.
55815.....	S	After 2 days.....	Fair.
55815.....	M	do.....	Do.
55816.....	S	do.....	Complete failure.
55816.....	M	do.....	Do.
55817.....	S	After 10 days.....	Fair.

^a Materials marked "L" were liquids at room temperature and were applied to the plates as such; materials marked "S" were semisolids and were smeared on the plates as a paste; in addition the semisolid materials were melted and applied to plates as liquids, and in such cases the mark "M" appears.

^b Ratings are given as follows: "Perfect," where the exposed steel plate shows no evidence of rust; "good," where the plate shows evidence of only a superficial and slight amount of rust; "fair," where the plate shows decided evidence of rust, but where a large percentage of the surface is still free from rust; "partial failure," where at least one-third of the surface of the plate is covered with rust; "complete failure," where the entire surface of the plate is covered with rust.

^c At the end of the 60 days' exposure this coating could not be readily removed by waste wet with kerosene.

TABLE 2.—Results of Exposure Tests for 60 Days upon 44 Samples of Rust Preventive Received from the War Department on July 11, 1919—Continued

B. S. laboratory No.	Materials	Appearance of first rust	Condition of plate after 60 days
55817.....	M	After 14 days.....	Fair.
55818.....	S	After 2 days.....	Partial failure.
55818.....	Mdo.....	Do.
55819.....	L	After 3 days.....	Complete failure.
55820.....	L	None.....	Perfect. ^a
55821.....	L	After 2 days.....	Complete failure.
55822.....	Ldo.....	Do.
55823.....	L	After 22 days.....	Fair. ^a
55824.....	L	After 27 days.....	Do. ^a
55825.....	L	After 22 days.....	Do. ^a
55826.....	L	After 2 days.....	Complete failure.
55827.....	Ldo.....	Do.

^a At the end of the 60 days' exposure this coating could not be readily removed by waste wet with kerosene.

TABLE 3.—Relation Between Ratings of Exposure and Results of Laboratory Tests upon 44 Samples of Rust Preventive from the War Department

Rating in exposure	Compounds	Passed all laboratory tests	Failed adhesion test	Failed slipping test	Failed corrosion test	Failed both adhesion and slipping tests	Failed both adhesion and corrosion tests	Film not removable with kerosene
Perfect.....	6	3	1	0	0	0	0	2
Good.....	3	1	0	0	0	1	0	1
Fair.....	13	2	2	3	1	3	2	0
Partial failure.....	10	1	1	3	0	5	0	0
Complete failure.....	19	2	4	1	5	2	3	2

Exposure tests on steel panels seemed to be a satisfactory method for distinguishing good slushing oils from poor ones, but this method was open to at least two very serious objections. In many cases the decision as to acceptance or rejection of rust preventives must be made in less than 60 days, and in some cases it would not be possible to await the results of an exposure test for a period of even 30 days. The results of exposure tests on rust preventives are influenced to a very great extent by weather conditions. Hot summer weather is a much more severe test upon a slushing oil than cold winter weather, and it is conceivable that a compound might be accepted upon the basis of 60 days exposure in winter, while the same material might be rejected from the results of summer exposure.

It was therefore seen to be very important that some laboratory tests be devised which would tell in a short time whether a given rust preventive would protect exposed metal under any conditions of weather, especially the severe conditions met with

during the summer near the seacoast with the accompanying salt-laden air. In some of the work already described it had been noticed that certain compounds seemed to be literally washed from the exposed steel plate by the first heavy rainfall. To distinguish this fatal defect in slushing oils, an artificial rain test was devised which will be described in detail further on.

It was considered that the failure of rust preventives which were not washed off the plate by rainfall was due to some one or more of three things: Either the material itself corroded metal, or it was more or less porous and allowed moisture to penetrate the film, or the rust preventive did not remain adherent to the metal owing to slippage or melting off of the coating by the heat of the sun.

It was believed that the detection of corrosive materials in the rust preventive itself was covered in an efficient manner by the corrosion test given in the original tentative specifications of January, 1919, and no change was made in this test.

A so-called oven salt-spray test was devised in which an attempt was made to simulate to a certain extent the combined conditions of hot weather and salt-laden air near the ocean. This new test was also designed to show up rust preventives which were porous or possessed the defect of slipping.

A specification for slushing oils was drawn up which embodied the new oven salt-spray test and the rain test. A comprehensive examination of a series of rust preventives was made according to these tests, and the specification was altered in certain details until the form given below was the final result.

VI. PROPOSED SPECIFICATIONS FOR SLUSHING GREASES SUITABLE FOR THE PROTECTION OF EXPOSED BRIGHT METAL

1. The material shall furnish a coating that shall firmly adhere to all metal surfaces at all temperatures at which they may be exposed under natural conditions.

2. The coating shall permanently remain in such a condition that it can be readily removed with cotton waste wet with kerosene.

3. CORROSION TEST.—When applied to polished iron, steel, brass, and copper surfaces and exposed at any temperature below 100° C (212° F) for a period of not less than five days, there shall be no stains on the metal or other evidence of corrosion due to the slushing oil.

4. OVEN SALT-SPRAY RAIN TEST.—Four bright steel plates approximately 4 by 6 inches shall be tacked to separate boards and thoroughly cleaned by the use of benzene. The slushing oil to be examined shall be melted, if not already liquid, and an excess flowed over the plates. One of the plates shall be kept in a vertical position in the laboratory for 24 hours and then placed in a rack on the roof so that the plate shall be inclined at an angle of 45° to the vertical, facing south. The plate shall be sprayed lightly with a 3 per cent salt solution on the first day. The other 3 plates shall be hung in a vertical position in an oven and maintained at a temperature of 45 to 50°C (113 to 122°F) for at least 48 hours. The plates shall be removed from the oven, allowed to cool, and 1 plate exposed on the roof in the rack described above. Another plate shall be kept in a horizontal position and lightly sprayed with a 3 per cent salt solution once every day for a period of 5 days. The fourth plate shall be placed under an intermittent shower, a vigorous shower being applied for approximately 3 to 4 minutes, then no water for about the same length of time. The shower is to be formed by allowing water to siphon at intervals from a 5-gallon tank into a metal trough, the bottom of which has three or four parallel rows of small holes (about one-sixteenth inch in diameter). The water should fall about 2 feet from the trough to the plate. This test should be continued for not less than 5 hours, the plate being held in a position about 60° to the vertical immediately under the falling water. At the end of 5 hours of this intermittent showering, the plate shall be placed in a horizontal position and allowed to remain with any adhering water for at least 24 hours. No rust shall be in evidence on any of the 4 plates after 5 days.

When time permits, the plates on the roof should be exposed for a period of 60 days, and at the end of this time there should be no appreciable rust in evidence.

DISCUSSION.—It is believed that any material which passes all the tests and requirements given above will protect bright exposed metal for a period of at least 60 days in any weather commonly encountered and will probably continue to protect exposed metal for many months. It has been found that the compounds which pass the above tests are usually greases of rather high melting point and should be applied to metal while melted, either by spraying or by dipping. We have seen a few samples that are liquid at ordinary temperatures which fulfill the requirements.

In order to gain some insight into the way in which the new laboratory tests on slushing oils would work out in routine examination of rust preventives, tests were made upon 23 of the 44 samples from the War Department previously referred to. Those compounds which had been found to corrode metal in the original tests were not selected. Compounds which had been found to give a varnish-like film were also left out. The results of exposure of each one of these 23 compounds during the most severe weather in Washington (July and August) was known. Each of these 23 compounds was carefully put through the oven salt-spray rain test outlined in the new proposed specification.

Seven of the 23 samples had been rated as "complete failure" in the 60-day exposure test. All 7 of these slushing oils failed conclusively in the oven salt-spray rain test. Seven of the 23 samples had been rated as "partial failure" in the exposure test. Six of these samples failed conclusively and 1 passed the oven salt-spray rain test. Four of the 23 samples had been given a rating of "fair" in the exposure test. All of these samples passed the oven salt-spray rain test. One of the 23 samples had been rated as "good" in the exposure test and it passed the oven salt-spray rain test. Four of the 23 samples had been rated as "perfect" in the exposure test. Three of these samples passed the oven salt-spray rain test, while the other sample showed slight evidence of rust on the edge of the plate.

From the above results it is seen that the laboratory tests in the new proposed specifications would exclude 13 out of 14 samples which had been rated as unsatisfactory in the exposure test. At the same time 1 sample out of 9 of the compounds rated as satisfactory in the exposure test would be excluded.

The results obtained above on the 23 samples by the laboratory tests in the new proposed specification are believed to average as well as most laboratory tests when they are compared with the results of actual service conditions or conditions similar to actual service.

Laboratory tests are almost never perfect, but it is believed that the tests outlined in the proposed specification are as good as any that can be given at the present time. Attention is called to the fact that even where it is possible to run exposure tests extending over considerable periods of time and some brand of material is found to give good service, rapid laboratory tests are still of great use in determining uniformity of quality of the product. It has often been noticed that different lots of the same

brand of rust preventive received at different times show wide variation in rust-preventing qualities. A rapid laboratory test can be used to determine the quality and uniformity of each shipment of slushing oil.

VII. GENERAL COMPOSITION OF SLUSHING OILS

During this investigation comparatively little work was done upon the composition of slushing oils. It was felt that it would be unwise to attempt in any specification for these materials to limit the manufacturer in the raw materials that he should use, because performance tests were more important than composition requirements.

Only a brief discussion of the general composition of commercial slushing oils will be given here, together with a description of some experimental batches of slushing oil prepared during the course of this investigation.

An examination of Table 1, on page 14, where the results of chemical tests upon 44 commercial samples of rust preventive are given, will give considerable insight into the composition of these samples. The majority of these materials showed a low acid number, together with a low ash content. These compounds are probably straight petroleum products of varying consistency from thin oils of low viscosity to petrolatum-like materials and thick petroleum residues.

The compounds which yield a considerable amount of ash may contain mineral matter as a filler (for example, one slushing oil seemed to be a mixture of lithopone and a heavy petroleum residue, while another seemed to be mainly iron oxide with a petroleum product), or they may be petrolatum-like compounds emulsified with a little water soluble chromate solution, supposed to act as a rust inhibitor.

The presence of small amounts of manganese with or without lead and calcium would indicate that some of the compounds are varnishes or mixtures of a petroleum product with varnish.

Several of the materials with high acid number were found to be blown vegetable oils. There were several black opaque compounds, which consisted of soft asphalt material or hard asphalt thinned with some solvent.

Rosin was detected in several of the compounds. In some cases it was present as an ingredient of varnish, while in other cases the slushing oil seemed to be a mixture of a petroleum product with rosin.

Among the 44 samples there was one, Bureau of Standards laboratory No. 55 814, which appeared to be a mixture of petrolatum and rosin, thinned with some kerosene. This compound was given a rating of perfect in the exposure test. A mixture based on the formula given below was made up in the laboratory in an attempt to duplicate the sample above mentioned.

20 g rosin, "H" grade,
100 g petrolatum (U. S. P.),
10 cc kerosene.

The rosin was melted and mixed with the previously heated petrolatum and the resulting material thoroughly stirred. After the mixture had partially cooled the kerosene was added. The presence of the rosin greatly increased the adhesive property of the mixture over that of the original petrolatum.

Bright steel plates were coated with this material in the usual way and exposed to the weather on September 9, 1919. On May 5, 1920, the plates were brought into the laboratory and cleaned off by means of cotton waste wet with kerosene. The film of rust preventive was readily removed and the surface of the steel underneath was found to be smooth and practically free from rust. The plates were nearly as bright as when they were coated and exposed eight months before.

The results of the above exposure test indicate that a good slushing oil can be made from ordinary petrolatum and rosin. Where a rust preventive might be exposed to extreme hot weather, such as a temperature of 120° F met with in the sun's direct rays, it was thought best to add something to the petrolatum-rosin mixture to raise its melting point.

The U. S. P. petrolatum used was smeared over the bulb of a thermometer and heated in an air bath. At 37° C. a drop of grease fell from the thermometer and this point was taken as the melting point of the material. Mixtures of rosin with petrolatum showed melting points little higher than the petrolatum alone. For example, 30 parts of rosin and 100 parts of petrolatum mixed together hot showed a melting point of 40° C by the above method. A wax, like carnauba, with a high melting point, was found to raise the melting point of petrolatum to a much more pronounced degree. For example, 40 parts of carnauba wax and 100 parts of petrolatum were mixed hot, and the melting point of the cold mixture found to be 79° C. In a similar way 40 parts of candelilla wax and 100 parts of petrolatum was found to melt at 59.6° C.

Two per cent of carnauba wax was found to raise the melting point of petrolatum to 52.5° C, 15.5 degrees higher than petrolatum alone.

Two formulas for a slushing grease of semisolid consistency are given below:

FORMULA A.—3 parts candelilla wax, 6 parts rosin, grade "H," 50 parts petrolatum (U. S. P.).

FORMULA B.—2 parts carnauba wax, 5 parts rosin, grade "H," 50 parts petrolatum (U. S. P.).

The above materials should be heated together at about 125° C until all the ingredients are melted and the mixture then thoroughly stirred and allowed to cool.

Both of the above mixtures were found to pass all the requirements for a slushing oil given in the proposed specification on page 18. The above mixtures were also applied to bright steel plates by flowing an excess of the melted grease over the surface. These plates were exposed to the weather and were found to be free from rust after several weeks' time.

The mixtures described under formula A and formula B are not claimed to be the best rust preventives which can be made to fulfill the requirements of the proposed specification, but are cited merely as examples of easily made compounds which were found to prevent rust in a satisfactory manner.

There are many manufacturers who employ different raw materials and make rust preventives of equal or better quality.

WASHINGTON, June 16, 1920.



